TITLE: PROCESSING, MICROSTRUCTURE AND CREEP BEHAVIOR OF MO-SI-B-BASED

INTERMETALLIC ALLOYS FOR VERY HIGH TEMPERATURE STRUCTURAL

**APPLICATIONS** 

**AUTHORS:** Vijay K. Vasudevan and Brian Riestenberg

**INSTITUTION:** The University of Cincinnati

Department of Chemical and Materials Engineering

501D ERC, Cincinnati, OH 45221-0012

**PHONE No.:** (513) 556-3103

**FAX No.:** (513) 556-3773

**E-MAIL:** vijay.vasudevan@uc.edu

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# PROGRAM INTRODUCTION: RATIONALE AND OBJECTIVES

This research project combines a novel processing, experimental and modeling approach, with detailed quantitative analysis of the influences of microstructure, in a basic study of the creep behavior of the next generation of refractory alloys based on the Mo-Si-B system. Through these studies, we will gain insight into the high-temperature creep behavior of these materials, including the effects of microstructure and the associated deformation, damage and failure features. A model Mo-rich Mo-Si-B alloy has been chosen for study, since it is representative of the new class of ductile-brittle systems, which, owing to their promising properties, are receiving evaluation for very high temperature structural applications. This project, which is closely coupled with ongoing activities at the Air Force Research Laboratory (AFRL) and Oak Ridge National Laboratory (ORNL) on these materials, focuses on three key areas to address issues related to the creep behavior: 1) basic materials processing and microstructural studies; 2) evaluation of the creep phenomenology and establishment of the constitutive behavior of the three-phase alloy and the individual phases within the alloy, including the effects of microstructure; and 3) theoretical modeling of the creep behavior based on analysis of associated creep data, creep mechanisms, damage processes and microstructural parameters. Microstructures will be analyzed in detail to provide quantitative information on aspects such as volume fraction, length scales, morphology and distribution of the second-phase intermetallics, the nature of their interfaces with the matrix, and the deformation, damage and failure processes during creep and this information coupled together with those of the creep properties and phenomenology to rationalize and theoretically model the observed creep behavior.

#### ACCOMPLISHMENTS DURING THE CURRENT PERIOD OF PERFORMANCE

This research project is concerned with developing a fundamental understanding of the effects of processing and microstructure on the creep behavior of refractory intermetallic alloys based on the Mo-Si-B system. These materials are representative of the next generation of refractory alloys being considered for very high temperature structural applications. During this period of performance, we have studied the microstructure and creep properties of a three-phase Mo-3 wt.% Si-1 wt.% B (Mo-8.9Si-7.71B in at.%) alloy that was produced using a powder metallurgy and supplied by UES/AFRL. Microstructural observations

using back-scattered electron imaging in a scanning electron microscope revealed that this allow was threephase, consisting of α-Mo, Mo<sub>3</sub>Si and T2-Mo<sub>5</sub>SiB<sub>2</sub> phases, with the combined volume fraction of the latter two being ~30%. Compression creep tests were conducted under inert atmosphere at 1200°C and 1100°C at stress levels ranging from 100 to 500 MPa in an Applied Test Systems creep frame equipped with computerized data acquisition. The results showed that the creep rates at 1200°C were quite high and the data of creep strain rate versus time or strain displayed two minima, one at small strains and the second at much larger strains. The former minima varied linearly from a value of 4.23 x 10<sup>-5</sup> min<sup>-1</sup> at 250 MPa to 7.95 x 10<sup>-4</sup> min-1 at 500 MPa, whereas the latter were 3.58 x 10-5 min-1 and 8.04 x 10-5 min-1 at the same stress levels, respectively. The stress exponent from the data corresponding to the first minima was determined to be ~4.26, which suggests that dislocation climb-glide creep dominates the creep process in the early stages. This value of the stress exponent is in the range of 2-7 reported for similar alloys by other investigators. The stress exponent determined from the second minimum creep rate data was ~1.18, which is near the value of 1 expected for diffusional creep and recrystallization mechanisms. At 1100°C, the minimum creep rates varied linearly with stress from 1.66x10<sup>-7</sup> min<sup>-1</sup> at 100 MPa to 2.13x10<sup>-6</sup> min<sup>-1</sup> at 300 MPa; the corresponding stress exponent was determined to be ~2.26, which suggests that both diffusional and dislocation mechanisms contribute to the overall creep process. The activation energy, Q, for creep was determined to be ~525 k]/mole, which is somewhat higher than the value of 400 k]/mole reported for self diffusion in α-Mo. Microstructural observations of post-crept samples indicated shape change and deformation of the α-Mo grains, together with the presence of many voids in these grains. A few cracks in the intermetallic particles and along their interfaces with the  $\alpha$ -Mo matrix were also observed. TEM observations of the crept samples revealed the presence of recrystallized α-Mo grains containing small-angle sub-grain boundaries made up of dislocation arrays. A few dislocations and sub-grains were also noted within the Mo<sub>3</sub>Si and T2 grains. These observations suggest that the bulk of the deformation and strain during creep is carried by the  $\alpha$ -Mo.

### **FUTURE WORK**

- Continue TEM analysis of microstructure and damage processes in the crept samples
- Conduct compressive creep testing of pure phase α-Mo and Mo<sub>3</sub>Si materials at selected temperatures and stresses matching those used for the three-phase alloy.
- Conduct compressive creep tests on Mo-Si-B alloys received from Dr. Joachim Schneibel at ORNL.
- Machine samples for tensile creep testing and complete this testing at selected temperatures and stresses.
- Complete modeling of creep in these alloys.

# LIST OF PAPERS PUBLISHED

"Compressive Creep Behavior of a Intermetallic-based Mo-3Si-1B Alloy," B. Riestenberg, M. G. Mendiratta, M. Scott and V. K. Vasudevan, *Intermetalics*, in preparation for submission (2006).

### LIST OF PRESENTATIONS

A paper based on the results obtained thus far was presented at the 2005 TMS/ASM Fall Meeting in Pittsburgh, PA in October (2005).

### STUDENTS SUPPORTED UNDER THIS GRANT

Brian Riestenberg and Bala Swaminathan, Ph.D. students, Dept. of Chemical and Materials Engineering, University of Cincinnati.